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Literature Review – Chip Seal Recycling

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Contact Details

Jeremy Wu

WSP
33 The Esplanade
Petone
Lower Hutt 5012
+64 4 587 0600
+64 21 241 3076
jeremy.wu@wsp.com

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Prepared by

Jeremy Wu, Lia van den Kerkhof, Phil Herrington

Reviewed by

Sheryl Tank, Lia van den Kerkhof

Approved for release by

Wendy Turvey

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1	Minor technical edits, definition of superheated and subcritical water added.
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Contents

Disclaimers and Limitations

This report ('**Report**') has been prepared by WSP exclusively for Waka Kotahi NZ Transport Agency ('**Client**') in relation to outline of the state of the art in chip seal recycling technologies ('**Purpose**') and in accordance with the Independent Professional Advisors (IPA) Contract NO 17-286 with the Client dated 22nd April 2021. The findings in this Report are based on interpretation of the literature in the subject areas. WSP accepts no liability whatsoever for any reliance on or use of this Report, in whole or in part, for any use or purpose other than the Purpose or any use or reliance on the Report by any third party.

1 Introduction

This is a preliminary investigation into current and emerging technologies for recycling of chip seal road surfacings. The intention of this research is to explore possible approaches for the development of a chip seal recycling practice comparable to the current practice of recycling reclaimed asphalt pavement (RAP).

According to the RAMM database, approximately 88% of the State Highway network is chip sealed the rest is surfaced with asphalt mix (except for a few kilometres in concrete). Assuming the proportion of chip seal surfacings for local authority roads is the same (it is probably higher) this gives approximately 57,000 km of chip seal surfacing throughout the country.

Chip seals are relatively simple to construct, consisting of a layer of bitumen, applied hot or as emulsion, onto which a layer of aggregate is spread and embedded. The bitumen acts as the ‘glue’ to hold the aggregates down, and the aggregates provide the texture and necessary friction for vehicles. Only certain aggregates are suitable (primarily due to skid resistance requirements), and these must often be trucked significant distances to where they are needed. Over time, a significant number of chip seal layer(s) build up, consisting of high quality bitumen (approximately 10-12% by weight) and aggregate. Much of the 57,000 km of chip seals consists of multiple (3 to 8) layers and is over 100 mm thick.

1.1 Current Practice

Currently, end-of-life chip seal surfacing is most commonly disposed of as clean-fill or buried under a new granular pavement layer as part of a rehabilitation treatment. In 2018-2019, in the Western Bay of Plenty District alone it has been estimated that over 40,000 m^2 of chip seal was milled to waste as part of the annual work programme. This represents about 600 tonnes of recoverable bitumen and 2,000 m^3 of quality aggregates.

In some cases the seal is milled up and mixed into the underlying granular material *in-situ*. This practice was introduced in the 1990s by researchers in the Hawkes Bay (Harrow 2008, Gray and Hart 2003) and has become an accepted practice in Australasia (Browne, 2020). The benefits of this practice largely lay in the savings associated with not transporting the seal material to waste and also some substitution of base course aggregate material. Ferguson (1989) reported the feasibility of recycling existing chip seal pavements through stabilisation with self-cementing fly ash. The amount of bitumen bound aggregate and fines influenced the degree of stabilisation but to a lesser degree than the amount of fly ash.

Neither treatment of old seal layers though makes best use of valuable bitumen and sealing chip resources but there is presently no recycling of chip seal surfacings undertaken in New Zealand.

Old asphalt mix surfacings are typically removed (milled), before new layers are applied to prevent the road surface becoming too high to fit gutters etc. RAP millings are frequently reused in new asphalt mix at about 10-30% by weight. Extensive research has been conducted on the use of RAP in manufacture of hot mix asphalt since the 1970s, due to the energy crisis which led to escalated bitumen prices, the use of RAP has become mainstream and a common practice in many road authorities across the world. RAP is now recognised as one of the most used recycled materials in the world by tonnage.

In contrast, the materials used in chip seals, which account for most New Zealand’s road surfacings are not being reused. Within the industry the potential recycling of chip seal surfacing has received only limited attention partly because of perceived problems relating to the higher and more variable bitumen content and aggregate composition compared to asphalt mix.

Preliminary work suggests that these problems are in fact not as significant as first thought and are discussed in more detail below.

1.2 Scale of the Resource

A conservative estimate of the quantity of bitumen available in the network can be made based on a 5m wide carriageway and 3 layers of single coat chip seal surface (at 1.5 Lm^{-2} residual application rate). As 88% of the 65,000 km sealed road network is chip seal (MoT statistics for 2019-2020), then there is 1,287,000,000 L of bitumen already in the country's road assets. This equates to $965,250,000 worth of bitumen (assuming a $750 per metric tonne of bitumen) as part of the road asset inventory. Assuming Grade 3 chip was used to construct the single coat seals, it is possible to estimate the amount of aggregate available in the multiple seal layers in orders of 14-16 million tonnes based on a nominal coverage rate of 135-160 m^2m^{-3} and a nominal aggregate density of 2.55 gcm^{-3}. According to Lane (2017), the New Zealand aggregate production figures from 2015 were approximately 39 million tonnes, 18 million of which were used in road construction.

2 Potential Approaches to Chip Seal Recycling

Chip seals could be recycled in two ways:

1. Incorporation of chip seal millings into an asphalt mix (i.e. equivalent to the recycling of RAP).
2. Recovery and reuse of the bitumen and aggregate components in either asphalt mix or chip seals.

2.1 Recycling of Chip Seal Millings

In principle, chip seal millings could be graded and incorporated, at a certain proportion (even 100%) into new asphalt mix in the same way as RAP. A concept proposed by researchers at WSP and Westlink was to *in-situ* recycle seal millings into an asphalt mix, using a small portable asphalt plant. Such plants are widely used overseas, typically make 1-3 tonnes of mix per hour and can be truck mounted. The mix would be used to replace or patch old seal surfaces in low demand, suburban streets or cul-de-sacs with low traffic levels. The quality of the mix could thus afford to be lower than that required under the NZTA M10 specification.

For successful recycling of seal surfaces into asphalt mix there are several technical issues that would need to be better understood:

2.1.1 Binder Content

Asphalt mix typically has 5-6 wt% of bitumen but levels in seals are much higher. Measurements of 144 seal core samples taken from the State Highway around the country (Herrington et al., 2015) has shown that the typical binder content is about 10 wt% (range of 7-11 wt%). These figures are consistent with calculations in Herrington et al. (2015) which show that based on standard seal design methods (Transit New Zealand, 2005) the expected bitumen content in a seal is 9 to 12 wt%. High bitumen contents can present problems with the particle size and stability of the millings (i.e. there is a tendency for agglomeration of stockpiles in hot weather or after long storage).

2.1.2 Binder Properties

In the New Zealand climate, oxidation of bitumen in chip seals is rapid over the first 2 years but slows markedly after that (Ball, 1999). Even so the properties of bitumen in seals along the road and within the layer of multi-layer seals will vary. This is also the case though with RAP and is allowed for through an initial investigation of the binder and if necessary, addition of rejuvenating agents.

One of the main inherent problems with RAP is the highly aged and thus stiff bituminous binder present in old asphalt mix. During the aging process, the maltenes / asphaltenes ratio in the bitumen decreases which leads to stiffening of the bitumen and ultimately to brittle behaviour (Moghaddam & Baaj, 2016). This is the main challenge when utilising high portions of RAP in the production of hot mix asphalt. To prevent premature fatigue and brittle failures of asphalt mix with RAP, binder rejuvenation via a rejuvenating agent is often required to restore the workability of the highly oxidised bitumen. The same principle would apply to the reutilisation of recovered bitumen from end-of-life chip seals discussed here.

Many studies have been conducted to investigate the effect of various rejuvenators including aromatic and naphthenic petroleum derived oils (most commonly used), various plant oils, waste vegetable oils and waste engine oils. (Moghaddam & Baaj, 2016, Zhang et al., 2019). Each type has different short-term and long-term effects on the subsequent asphalt mixture, but the main purpose of a rejuvenating agent is to restore the maltenes / asphaltenes ratio in the bitumen (not just to plasticise the bitumen through solvent action). Rejuvenators are normally added in at about 5% to 10% but sometimes up to 20% by weight. Therefore, the environmental impact of the (petroleum based) rejuvenator itself can be significant. In recent years, to solve this problem, "bio-rejuvenators" derived from renewable plant sources have been the subject of growing research.

The review conducted by Fang et al. (2021) shows that the bio-rejuvenators can significantly improve the low-temperature performance of aged bitumen and restore its high-temperature performance to the level consistent with the virgin material. To date, waste oil based rejuvenators (waste soybean oil, waste vegetable oil, and waste cooking oil) have dominated the research area followed by products derived from the pyrolysis of wood-fibre or animal manure.

The use of renewable plant based rejuvenators produced locally has the advantage of potentially lowering the carbon footprint of a chip seal recycling process.

2.1.3 Aggregate Grading

The grading of aggregates in chip seals is obviously very different from asphalt mix at the time of construction, consisting of mainly grade 5 to grade 2 size chips specified by the Waka Kotahi M6 specification (roughly 3-5 mm to 13 mm). After a few years' service however the grading is much more continuous due to aggregate wear (see Figure 1). The grading will vary depending on the seal age and design (as is the case with RAP). Data from seal cores indicates recycling using an open-graded porous asphalt mix design may be most appropriate (see the Field Investigation below).

Figure 1: Aggregate extracted from a multi-layer chip seal core showing aggregate wear.

2.1.4 Field Investigation

A number of cores were taken from SH36 in 2013 as part of a preliminary study on chip seal recycling by WSP and Downer (Road Science). The object of the exercise was to get an appreciation of likely variability in a "randomly" selected section of State Highway. Binder contents and gradings were determined. The binder from some cores was extracted and the viscosity determined.

A total of 38 cores were taken over about 1.5 km from the outer wheelpath on both left and right lanes. At most of the core locations there were 3 seal layers but some were only 1 or two layers thick. The thickness of the seals ranged from 13 to 70 mm, with a mean of 35 mm (see Figure 2).

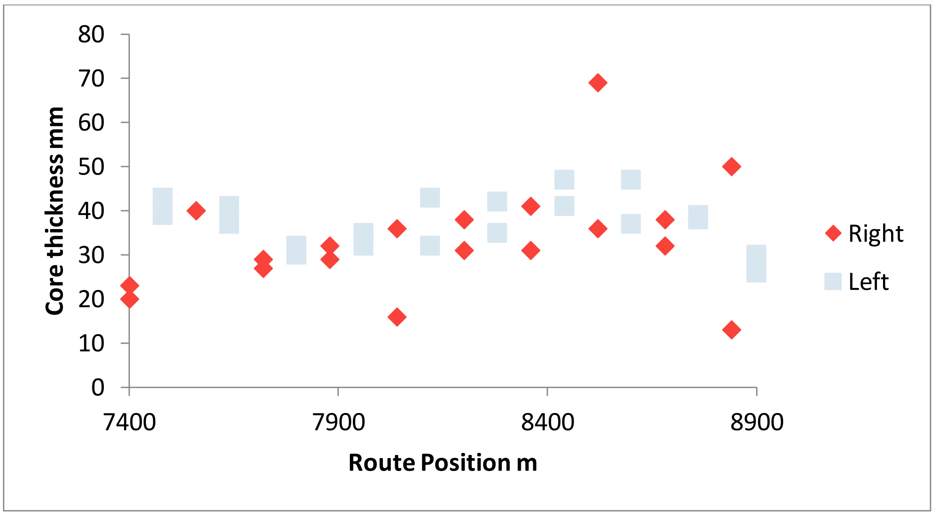

Figure 2: Chip seal core depths from SH36 investigation (right and left lanes).

The binder content, grading and bitumen properties of selected cores were measured. The position of the cores and seal history (from RAMM) at each location are shown in Table 1. Unfortunately, the age of the seals from which these cores were taken was not determined at the time of the study.

Table 1: Core samples analysed from SH36 in 2013.

Core number	Chainage (m)	Seal layer 1 chip grade	Seal layer 2 chip grade	Seal layer 3 chip grade
9	7720	4	4	-
11	7800	3	4	4
13	7880	4	4	5
14	7880	3	4	5
17	8040	3	4	5
19	8120	3	4	5
21	8200	3	3	4
23	8280	3	3	3
24	8280	3	3	5
25	8360	3	3	4
27	8440	3	3	3

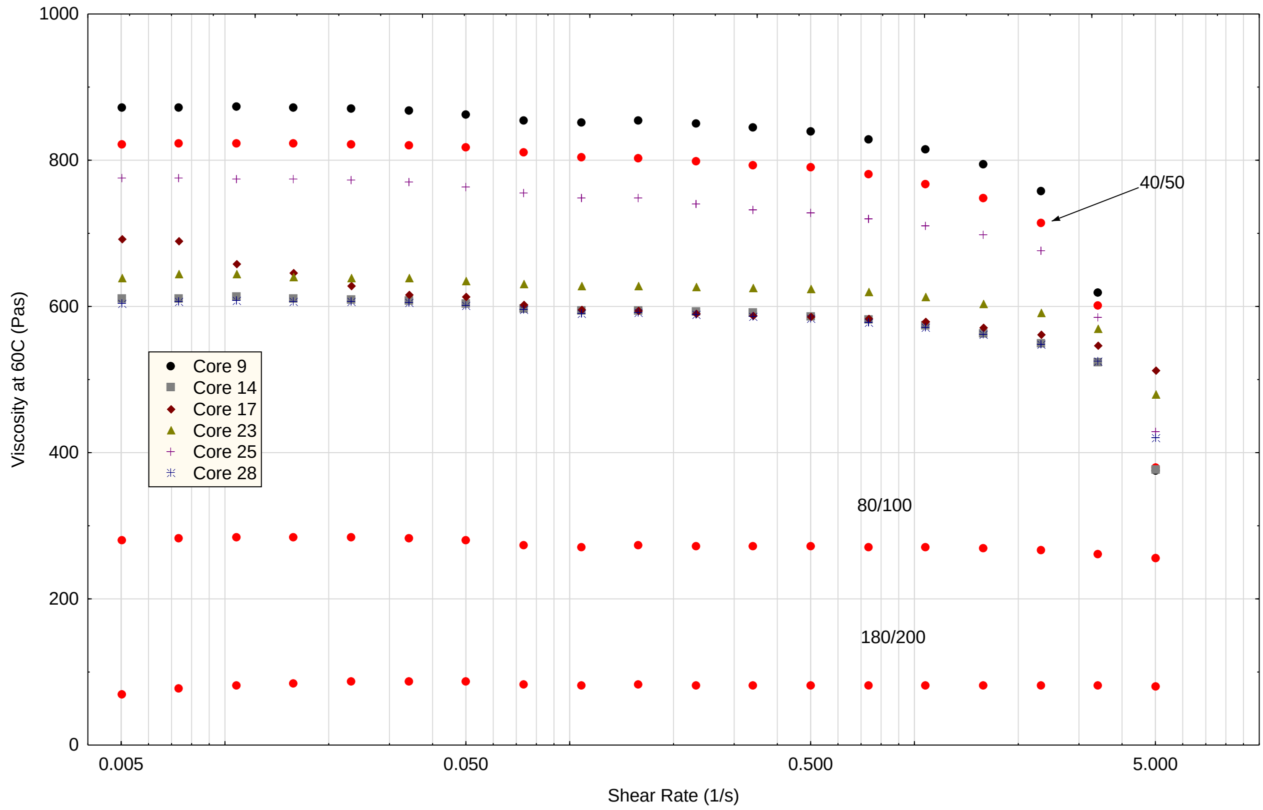

Figure 3: Viscosity at 60 °C of extracted bitumen compared to standard bitumen grades.

The viscosity of the bitumen (extracted using dichloromethane and an in-house method) was measured using a cone and plate viscometer. The absence of extraction solvent was confirmed by FTIR spectroscopy. The results are shown in Figure 3 and compared to the viscosity of standard NZTA M1 grades of bitumen.

Table 2: Aggregate gradings for randomly selected cores.

Core #	11	13	19	21	24	27	Mean
Sieve size:	Percent passing						
19.0 mm	100	100	100	100	100	100	100
16.0 mm	100	100	99	100	98	100	100
13.2 mm	96	99	95	98	93	94	96
9.5 mm	53	52	55	53	51	55	53
6.7 mm	27	31	31	30	30	32	30
4.75 mm	17	21	19	17	17	17	18
2.36 mm	13	15	13	12	12	11	13
1.18 mm	10	12	10	10	10	8	10
600 µm	8	11	8	8	8	7	8
300 µm	7	9	6	6	7	5	7
150 µm	5	7	5	5	5	4	5
75 µm	4	6	3	3	4	3	4
Binder content (%)	10.5	9.9	10.7	10.6	10.5	10.2	10.4

The extracted binders had hardened from the initial 130-150 grade bitumens that would have been used in the construction but showed a relatively small range. The approximate penetration at 25 °C was estimated from log-log plot of penetration versus viscosity (at 0.01 s^{-1}). These ranged from 44 to 55.

The gradings of six of the cores are shown in Table 2 and plotted in Figure 4. The gradings are compared to the grading envelope for porous asphalt (PA14) from the NZTA P11 specification. The core gradings fall largely within the PA grading except for the 9.5 mm sieve.

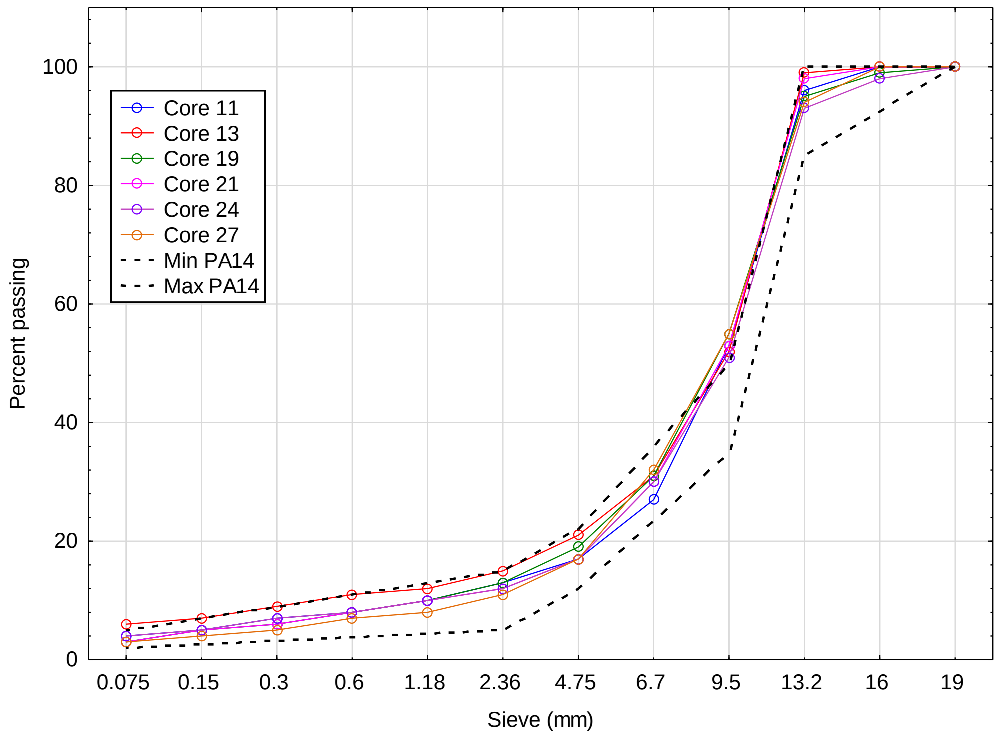

Figure 4: Aggregate grading curves showing PA 14 specification envelope (dotted lines).

2.2 Recovery and Reuse of Bitumen and Aggregate

While encouraging the use of chip seal millings would improve the current recycling rate of end-of-life road surfacing, it is much more attractive to separate the millings into aged bitumen and 'clean' aggregate as it would provide the industry more options to utilise the recovered resources.

The following section covers literature on the methods used in extracting bitumen from aged road surfacings with the intention to separate the bitumen and the aggregate as individual resources. These methods are largely at the research stage or used solely for quality control testing. Although patents relating to asphalt mix and roofing shingles exist (e.g. Robinson et al., 1987, Nykulin,et al., 2012) there does not appear to be any widespread uptake of recovery technologies at the industrial scale.

2.3 Extraction and Recovery Methods

2.3.1 Organic Solvent Extraction Methods for Recovering Bitumen

Bitumen is soluble in many organic solvents and there are many established laboratory-based extraction and recovery methods for recovering bitumen from bituminous mixtures (Ziyani et al., 2017). For example is ASTM D2172 Method A: high-speed centrifuge extraction; Method B: reflux extraction; Method C: vacuum extraction; and Method D: extraction kettle.

Once the bitumen is extracted, there are several recovery methods available to remove the solvent including the Abson method (ASTM D1856) which is a distillation method, the rotary evaporation method (ASTM D5404), and the modified D5404 method with the use of toluene as a solvent (ASTM D7906). Toluene has a higher boiling point of 110.6 °C when compared to the solvents used in D5404 (87 °C for trichloroethylene, 71 °C for n-propylbromide, 39.8 °C for dichloromethane).

The American Association of State Highway and Transportation Officials (AASHTO) approved T319 as a method which combines the principles of the above methods and has been demonstrated to recover aged bitumen with minimal amount of hardening by the solvent.

All of the above, and many similar methods, are mainly used for quality control testing. Scale up of a solvent extraction and recovery process using a low-boiling solvent such as dichloromethane would be in principal relatively simple (Robinson et al., 1987). The solvent would be recovered and recycled indefinitely in the process. Terpene based or other solvents from renewable sources could be used in place of petroleum derived materials. Complete removal of aggregate fines in the sub 100-micron range would probably not be feasible, but neither would it be necessary.

2.3.2 Hydrothermal Recovery

Amoussou et al. (2016) reported on an interesting method to recover bitumen from reclaimed asphalt pavement (RAP) using a combination of pulsed power discharge technology and hydrothermal water. The process is based on previous work done by the same authors (Amoussou et al., 2015, Inoue et al., 2009) using pulsed power discharge technology to recover useful products from construction sector wastes such as concrete in Japan.

Pulsed power discharge technology is defined as a process in which energy is accumulated over a relatively long time period and then discharged in short pulses in a controlled rate and thus the amount of power in each pulse is concentrated. It is claimed to consume less energy overall than conventional crushing processes. Such technology has been developed for applications such as food processing, exhaust gas treatment, water treatment, engine ignition, electromagnetic pulse generators and other advanced industrial applications (Takaki & Katsuki, 2009). Nevertheless, it does not appear to be widely taken up in the recycling industry, possibly due to the high upfront capital costs.

In the 2016 study, Amoussou et al. used subcritical water to investigate the effect of hydrothermal process variables (water ratio, temperature, stirring speed, treatment time) on the recovery rate and material properties of the recovered product. Subcritical water refers to liquid water at temperatures between atmospheric boiling point and the critical temperature of water (374°C), and superheated water refers to liquid water above the critical temperature. These are relatively harmless solvents and have garnered research interest due to the growing interest in green chemistry (Smith, 2006) since water is abundant, cheap, and easily recyclable. However supercritical water has been proven to be an unsuitable solvent because the extract, the bitumen in this case, is easily oxidised.

Elemental analysis of binder extracted from RAP indicated that apart from a higher oxygen content due to normal field aging, the recovered bitumen has similar chemical composition to the virgin bitumen. As expected, the recovered bitumen had hardened and had a lower ductility due to oxidation. Therefore, the recovered bitumen would need to be rejuvenated somehow before being reutilised as a binder that meets performance requirements.

In terms of the optimum hydrothermal condition, it was reported that a water:RAP ratio of 2:1 (2 mLg^{-1}), temperature of 300 °C, stirring speed of 450 RPM, and treatment time to up to 3 hours gave the best bitumen recovery yield. The authors further experimented with cooling conditions and found that stirring during cooling assisted with improving the 'cleanliness' of the recovered aggregates but did not disclose the exact procedure. Due to the limitation of the equipment, the authors have noted that further work is needed to investigate the effect of temperatures beyond 300 °C.

While there may be a high upfront cost to access pulsed power technology, the hydrothermal method, in principle, presents a technically viable route to separate aged bitumen from a bituminous mix that does not involve organic solvents. Using water as a recovery solvent also takes advantage of the natural affinity of water to polar aggregate surfaces and its tendency to disbond bitumen.

Kano et al. 2016 used water at temperatures of 300 to 450 °C and pressures of 20-45 MPa showed that supercritical water could effectively separate bitumen and aggregate in RAP samples. In a more recent study, Akatsu et al. (2020) also suggested the potential of using hydrothermal methods as a recovery technology for aged bitumen. That study investigated the effectiveness of subcritical water on aged bitumen based on evaluation of the physical properties, chemical properties, and dynamic rheology. Asphalt mix reacted for 15 minutes at 350 to 360 °C showed a significant recovery yield and modifying effects on physical properties such as penetration, softening point and ductility. Similar effects were found in bitumen with a different composition. In addition, the chemical properties confirmed that bitumen recovered by the hydrothermal decomposition method has lower molecular weight and reduced oxidation. Bitumen after hydrothermal decomposition had improved fatigue crack resistance and flow resistance compared to virgin asphalt, indicating the potential of hydrothermal decomposition for recycling asphalt mixtures.

A drawback of high pressure methods is that scale up to industrial scales can be very expensive. Using temperatures of above 350 °C will also lead to decomposition (cracking) of the bitumen to some degree. This may actually be of benefit though as lower molecular weight material will act to plasticise the hardened bitumen.

2.3.3 Recovery of Bitumen from Oil Sands

Large deposits of heavy oil/natural bitumen oil sands exist world-wide. Major deposits are found in Canada in particular with typically about 10% bitumen content. The hydrocarbon material in these deposits is referred to as bitumen although it differs somewhat from refined petroleum bitumen used in road construction, especially in having a lower viscosity (it has the consistency of cold molasses). Extraction of the bitumen from oil sands has been an area of research since the early 20th century. The technology developed for that purpose may have application in recovery of bitumen from chip seals.

There are various commercial processes currently in use, many of which use water (typically at 45-55 °C) as the primary extractant (Masliyah et al., 2004). The conditions used are very mild compared to those discussed in section 2.3.2. The sands are initially agitated with water to breakup lumps and disbond the bitumen from the aggregate material. Bitumen droplets are floated to the surface using air which is bubbled through the slurry. This is a key component of the process as it speeds the separation which would otherwise be too slow (bitumen density is very close to that of water). The bitumen-rich froth is removed. To further refine the material, which still contains about 10% fine solids, solvent (e.g. naptha or hexane) is added. This lowers the bitumen density to aid in the separation from residual water in the froth. The resulting oil/solvent mixture is then piped offsite for refining. The water used is recycled.

Adaptation of this type of technology may be successful with chip seal millings although an obvious difference is that the bitumen in the millings will have a much high viscosity than that of oil sand bitumen. On the other hand, the efficiency of oil sand bitumen extraction is adversely affected by the presence of naturally occurring fines (Williams et al., 2010). These are present at about 15-30% in the sub 44 micron range, much higher than that in chip seal millings. Much of the fines are clays which are particularly difficult to separate from bitumen droplets. These would be largely absent in chip seal millings.

3 Discussion

There is a very large bitumen and aggregate resource in our chip seal surfacings which is currently not being recycled or reused effectively. Recycling of chip seal surfacings would reduce the volume of material going to cleanfill and the demand for non-renewable virgin aggregates and bitumen.

Two approaches can be taken to recycling. The first, relatively simple in principal, is to emulate the use of RAP and incorporate chip seal millings into asphalt mix (probably an OGPA type). The second, more ambitious route is to develop technology to recover the bitumen and aggregate seal components separately.

In the short to medium term, recycling of chip seals as an additional RAP-like resource may be the most practicable engineering solution. In the long run, from a resource efficiency point of view, recycling of chip seals, by separating the materials, would make a greater impact on resource utilisation given that amount of bitumen and aggregate materials that would potentially be made available from the recovery process. This would reduce the demand for quarrying of virgin aggregate and importation of bitumen (there is no local manufacturing of bitumen). The upcoming challenges in the bitumen supply market is discussed further in a separate report "Bitumen Alternatives" prepared by Herrington et al. (2021) on behalf of Waka Kotahi.

It is estimated that between 150-640 kg of CO_2e is generated per tonne of bitumen manufactured offshore (Herrington et al., 2021). This figure varies due to transport distances, crude oil source, ship capacity, etc. Nevertheless, if it is possible to extract and recover bitumen from chip seal surfaces, and using plant based rejuvenators, then it is entirely plausible that a significant amount of CO_2 generated from the processing of crude oil and the transportation (importation) of bitumen into New Zealand could be eliminated. Similarly, the carbon footprint of aggregate production could potentially be reduced if aggregate recovered from chip seals can be used instead of adding further pressure to the already-stretched virgin aggregate supply.

Both recycling methods discussed in this report require research to establish their technical, economic and environmental viability. Some field work has already been undertaken in New Zealand with chip seal millings that gave very promising results.

4 Recommendations

Given that there is little published work on recycling of chip seal surfaces, it is recommended that further research is undertaken in the following areas:

1. Utilisation of chip seal millings in asphalt pavements via small-scale *in-situ* recycling and placement of asphalt (or *ex-situ* processes similar to RAP). This would potentially have a short-term, immediate impact on the recycling rate of chip seal surfaces;
2. Extraction and recovery processes for separation of bitumen and aggregate from chip seal millings which can be scaled up, and are commercially and environmentally viable;
3. Life Cycle Assessment sensitivity studies of the new processes conceptualised in (1) and (2) above to help understand the constraints required for a viable process. The LCA studies would use RAP methods as a benchmark for comparisons;
4. Potential sustainable, environmentally friendly, rejuvenating agent from renewable materials for modifying the extracted, aged bitumen to enable its use in both asphalt and chip seals.

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